PURIFICATION OF LABORATORY CHEMICALS

Fourth Edition

W.L.F Armarego
Pteridine Biochemistry Laboratory
Protein Biochemistry Group
Division of Biochemistry & Molecular Biology
The John Curtin School of Medical Research
Australian National University, Canberra
A.C.T

And

D.D Perrin
Formerly of the Medical Chemistry Group
Australian National University, Canberra
A.C.T

BUTTERWORTH HEINEMANN

Butterworth-Heinemann
Linacre House, Jordan Hill, Oxford OX2 8DP
225 Wildwood Avenue, Woburn, MA 01801-2041
A division of Reed Educational and Professional Publishing Ltd

A member of the Reed Elsevier plc group

OXFORD AUCKLAND BOSTON
JOHANNESBURG MELBOURNE NEW DELHI

First published 1996
Paperback edition 1997
Reprinted 1998, 1999, 2000

British Library Cataloguing in Publication Data
A catalogue record for this book is available from the British Library

Library of Congress Cataloguing in Publication Data
A catalogue record for this book is available from the Library of Congress

ISBN 0 7506 3761 7

Printed and bound in Great Britain by The Bath Press, Bath

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nitrogen and passed through activated alumina before use. [Woon et al. *JACS* **108** 7990 *1986*; Wong et al. *JACS* **109** 3428 *1987*].

Dimethylcarbamoyl chloride *[79-44-7]* **M 107.5, m -33°, b 34°/0.1mm, d 1.172, n 1.4511.** Must distil under high vacuum to avoid decomposition.

3,3'-Dimethylcarbanilide *[620-50-8]* **M 240.3, m 225°.** Crystd from ethyl acetate.

Dimethyl carbonate *[616-38-5]* **M 90.1, m 4.65°, b 90-91°, d 1.070, n 1.369.** Contains small amounts of water and alcohol which form azeotropes. Stood for several days in contact with Linde type 4A molecular sieves, then fractionally distd. The middle fraction was frozen slowly at 2°, several times, retaining 80% of the solvent at each cycle.

***cis-and trans*-1,4-Dimethylcyclohexane** *[589-90-2]* **M 112.2, b 120°, d 0.788, n 1.427.** Freed from olefines by shaking with conc H_2SO_4, washing with water, drying and fractionally distilling.

5,5-Dimethyl-1,3-cyclohexanedione see **dimedone.**

1,2-Dimethylcyclohexene *[1674-10-8]* **M 110.2, b 135-136°/760mm, d 0.826, n 1.4591.** Passed through a column of basic alumina and distd.

1,5-Dimethyl-1,5-diazaundecamethylene polymethobromide (Hexadimethrene, polybrene) *[28728-55-4]*. Purified by chromatography on Dowex 50 and/or by filtration through alumina before use [Frank *Hoppe-Seyler's Z Physiol Chemie* **360** 997 *1979*].

Dimethyldihydroresorcinol see **dimedone**.

2,9-Dimethyl-4,7-diphenyl-1,10-phenanthroline *[4733-39-5]* **M 360.5, m >280°.** Purified by recrystn from benzene.

Dimethyl disulphide *[624-92-0]* **M 94.2, f.p. -98°, b 40°/12mm, 110°/760mm, d 1.0605, n 1.5260.** Passed through neutral alumina before use.

Dimethyl ether see **methyl ether.**

2,2-Dimethylethyleneimine *[2658-24-4]* **M 71.1, b 70.5-71.0°.** Freshly distd from sodium before use.

***N,N*-Dimethyl formamide (DMF)** *[68-12-2]* **M 73.1, b 76°/39mm, 153°/760mm, d 0.948, n^{25} 1.4269.** Decomposes slightly at its normal boiling point to give small amounts of dimethylamine and carbon monoxide. The decomposition is catalysed by acidic or basic materials, so that even at room temperature DMF is appreciably decomposed if allowed to stand for several hours with solid KOH, NaOH or CaH_2. If these reagents are used as dehydrating agents, therefore, they should not be refluxed with the DMF. Use of $CaSO_4$, $MgSO_4$, silica gel or Linde type 4A molecular sieves is preferable, followed by distn under reduced pressure. This procedure is adequate for most laboratory purposes. Larger amounts of water can be removed by azeotropic distn with benzene (10% v/v, previously dried over CaH_2), at atmospheric pressure: water and benzene distil below 80°. The liquid remaining in the distn flask is further dried by adding $MgSO_4$ (previously ignited overnight at 300-400°) to give 25g/L. After shaking for one day, a further quantity of $MgSO_4$ is added, and the DMF distd at 15-20mm pressure through a 3-ft vacuum-jacketed column packed with steel helices. However, $MgSO_4$ is an inefficient drying agent, leaving about 0.01M water in the final DMF. More efficient drying (to around 0.001-0.007M water) is achieved by standing with powdered BaO, followed by decanting before distn, with alumina powder (50g/L; previously heated overnight to 500-600°), and distilling from more of the alumina; or by refluxing at 120-140° for 24h with triphenylchlorosilane (5-10g/L), then distilling at *ca* 5mm pressure [Thomas and Rochow JACS 79 1843 1957]. Free amine in DMF can be detected by colour reaction with 1-fluoro-2,4-dinitrobenzene. It has also been purified by drying overnight over KOH pellets and then distd

from BaO through a 10 cm Vigreux column {*Experimental Cell Research* **100** 213 *1976*]. [For efficiency of desiccants in drying dimethyl formamide see Burfield and Smithers [*JOC* **43** 3966 *1978*, and for a review on purification, tests of purity and physical properties, see Juillard *PAC* **49** 885 *1977*].
It has been purified by distilling from K_2CO_3 under high vac and fractionated in an all-glass apparatus. The middle fraction is collected, degassed (seven or eight freeze-thaw cycles) and redistd under as high a vacuum as possible [Mohammad and Kosower *JACS* **93** 2713 *1971*].

***d,l*-2,4-Dimethylglutaric acid** *[2121-67-7]* **M 160.2, m 144-145°.** Distd in steam and crystd from ether/pet ether.

3,3-Dimethylglutaric acid *[4839-46-7]* **M 160.2, m 103-104°, b 89-90°/2mm, 126-127°/4.5mm.** Crystd from water, benzene or ether/pet ether. Dried in a vacuum.

3,3-Dimethylglutarimide *[1123-40-6]* **M 141.2, m 144-146°.** Recrystd from EtOH [Arnett and Harrelson *JACS* **109** 809 *1987*].

***N,N*-Dimethylglycinehydrazide hydrochloride** *[539-64-0]* **M 153.6, m 181°.** Crystd by adding EtOH to a conc aqueous soln.

Dimethylglyoxime *[95-45-4]* **M 116.1, m 240°.** Crystd from EtOH (10ml/g) or aqueous EtOH.

2,5-Dimethyl-2,4-hexadiene *[764-13-6]* **M 110.2, f.p. 14.5°, b 132-134°, d 0.773, n 1.4796.** Distd, then repeatedly fractionally crystd by partial freezing. Immediately before use, the material was passed through a column containing Woelm silica gel (activity I) and Woelm alumina (neutral) in separate layers.

2,2-Dimethylhexane *[590-73-8]* **M 114.2, m -121.2°, b 107°, d 0.695,**
2,5-Dimethylhexane *[592-13-2]* **M 114.2, m -91.2°, b 109°, d 0.694.** Dried over type 4A molecular sieves and distd.

2,5-Dimethylhexane-2,5-diol *[110-03-2]* **M 146.2, m 88-90°.** Purified by fractional crystn. Then the diol was dissolved in hot acetone, treated with activated charcoal, and filtered while hot. The soln was cooled and the diol was filtered off and washed well with cold acetone. The crystn process was repeated several times and the crystals were dried under a vac in a freeze-drying apparatus [Goates et al. *JCSFT 1* **78** 3045 *1982*].

5,5-Dimethylhydantoin *[77-71-4]* **M 128.1, m 177-178°.** Crystd from EtOH and sublimed *in vacuo*.

1,1-Dimethylhydrazine *[57-14-7]* **M 60.1, b 60.1°/702mm, d 0.790, n 1.408.** Fractionally distd through a 4-ft column packed with glass helices. Ppted as its oxalate from ethyl ether soln. After crystn from 95% EtOH, the salt was decomposed with aqueous saturated NaOH, and the free base was distd, dried over BaO and redistd [McBride and Kruse *JACS* **79** 572 *1957*]. Distn and storage should be under nitrogen.

4,6-Dimethyl-2-hydroxypyrimidine *[108-79-2]* **M 124.1, m 198-199°.** Crystd from absolute EtOH (charcoal).

1,2-Dimethylimidazole *[1739-84-0]* **M 96.1, b 206°/760mm, d 1.084.** Crystd from benzene and stored at 0-4°. [Gorun et al. *JACS* **109** 4244 *1987*].

1,1-Dimethylindene *[18636-55-0]* **M 144.2.** Purified by gas chromatography.

Dimethyl itaconate *[617-52-7]* **M 158.2, m 38°, b 208°, d 1.124.** Crystd from MeOH by cooling to -78°.

Dimethylmaleic anhydride *[766-39-2]* **M 126.1, m 96°, b 225°/760mm.** Distd from benzene/ligroin and sublimed in a vacuum.